



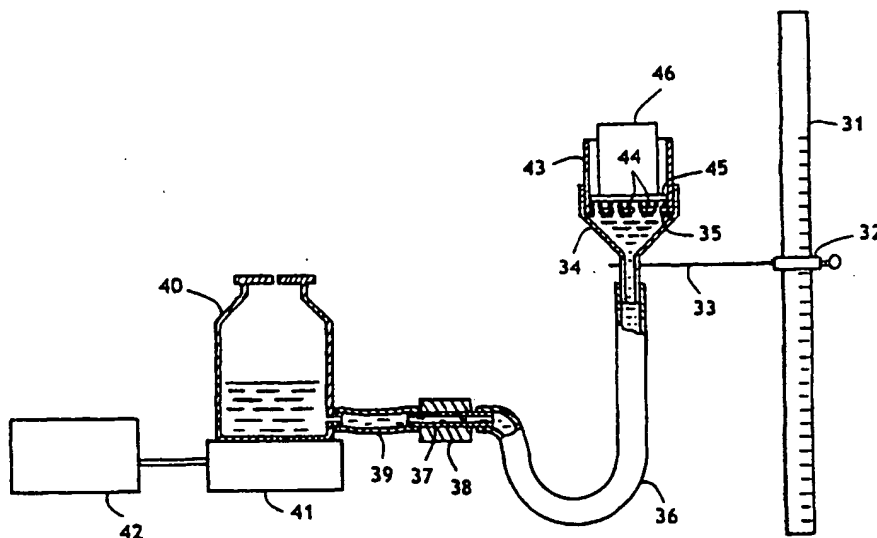
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(54) Title: **ADSORBENTS FOR USE IN HANDLING COMPLEX FLUIDS**



(57) Abstract

Adsorbent particles exhibiting an efficacy in the handling of complex fluids. Particles of adsorbent material demonstrating an efficacy in the handling of complex fluid are suitable for incorporation into disposable absorbent articles and the like.

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ADSORBENTS FOR USE IN HANDLING COMPLEX FLUIDS

BACKGROUND

The present invention relates to the use of adsorbent particles in absorbent
5 structures and disposable absorbent articles. More particularly, the present invention
relates to adsorbent particles exhibiting an efficacy in the handling of complex fluids.

The use of adsorbent particles in disposable absorbent articles is known. Such
adsorbent particles are generally employed on a somewhat limited basis for odor control
in disposable personal care absorbent articles such as feminine hygiene products and the
10 like. However, a more widespread use of such adsorbent particles in absorbent structures
and disposable absorbent articles has been somewhat confined by the limited efficacy of
the adsorbent particles in the handling of complex fluids. Thus, it would be desirable to
improve the efficacy of adsorbent particles in the handling of complex fluids, potentially
resulting in the broadened use of such adsorbent particles in absorbent structures and
15 disposable absorbent articles.

SUMMARY

The present inventors have recognized the difficulties and problems inherent in
the prior art and in response thereto conducted intensive research into developing
20 adsorbent particles that exhibit an efficacy in the handling of complex fluids. While
conducting such research, the inventors found that certain adsorbent materials exhibit an
efficacy in the handling of complex fluids. The efficacy of these adsorbent materials can
be improved by the appropriate selection of a suitable pore size distribution. As a result
of this improved efficacy, the adsorbent materials of the present invention allow for the
25 broadened use of adsorbent particles in absorbent structures and disposable absorbent
articles.

In one embodiment of the present invention, an absorbent articles includes a
containment means and particles of at least one adsorbent material. While in the
containment means, about 20 to about 50 percent of the adsorbent particles have a pore
30 volume coming from pores greater than about 100 microns for complex fluid intake and
complex fluid distribution. About 80 to about 50 percent of the pore volume of the

adsorbent particles comes from pores smaller than about 100 microns for complex fluid retention. The adsorbent particles are present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

5 In another embodiment of the present invention, an adsorbent article includes a containment means and particles of at least one adsorbent material. While in the containment means, about 10 to about 100 percent of the adsorbent particles have a permeability of at least about 1,000 K. The adsorbent particles are present in the containment means in an amount of from about 10 to about 100 percent based on the total
10 weight of the containment means and the adsorbent particles.

 In still another embodiment of the present invention, an adsorbent article includes a containment means and particles of at least one adsorbent material. The adsorbent particles in the containment means have a complex fluid retention capacity of at least 2 g/g. The adsorbent particles are present in the containment means in an amount of from
15 about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

 In a further embodiment of the present invention, an adsorbent article includes a containment means and particles of at least one adsorbent material. The adsorbent particles in the containment means have a minimum average particle size of at least about
20 200 microns with a standard deviation of at least about 25 percent of the average particle size. The adsorbent particles are present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

 In yet another embodiment of the present invention, an adsorbent article includes
25 a containment means and particles of at least one adsorbent material. The adsorbent particles in the containment means have a multi-modal particle size distribution and are present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

DRAWINGS

These and other features, aspects and advantages of the present invention will become better understood with regard to the following description, appended claims and accompanying drawings where:

5 FIG. 1 is an illustration of an apparatus suitable for measuring the pore size distribution of an adsorbent material using the capillary tension method.

FIG. 2 is an illustration of an apparatus suitable for determining the Gel Bed Permeability (GBP) value of an adsorbent material.

FIG. 3 is an illustration of an overhead view of the piston head of the apparatus in
10 FIG. 2.

DESCRIPTION

The adsorbent materials of the present invention include adsorbent particles and adsorbent particles that are treated with a surface modifying agent. By "particle,"
15 "particles," "particulate," "particulates" and the like, it is meant that the adsorbent material is generally in the form of discrete units. The particles can comprise granules, pulverulents, powders or spheres. Thus, the particles can have any desired shape such as, for example, cubic, rod-like, polyhedral, spherical or semi-spherical, rounded or semi-rounded, angular, irregular, etc. Shapes having a large greatest dimension/smallest
20 dimension ratio, like needles, flakes and fibers, are also contemplated for use herein. The use of "particle" or "particulate" may also describe an agglomeration comprising more than one particle, particulate or the like and may also consist of more than one type of adsorbent.

As used herein, the term "nonwoven" refers to a web that has a structure of
25 individual fibers or filaments which are interlaid, but not in an identifiable repeating manner.

As used herein, the terms "spunbond" or "spunbonded fiber" refer to fibers which are formed by extruding molten thermoplastic material as filaments from a plurality of fine, usually circular, capillaries of a spinneret with the diameter of the
30 extruded filaments then being rapidly reduced.

“Coform” as used herein is intended to describe a blend of meltblown fibers and cellulose fibers that is formed by air forming a meltblown polymer material while simultaneously blowing air-suspended cellulose fibers into the stream of meltblown fibers. The meltblown fibers containing wood fibers are collected on a foraminous belt
5 that may have placed onto its surface a pervious material such as spunbonded fabric material.

As used herein, the phrase “meltblown fibers” refers to fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into a high velocity, usually heated, gas (e.g.,
10 air) stream which attenuates the filaments of molten thermoplastic material to reduce their diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a web of randomly disbursed meltblown fibers.

As used herein, the phrase “complex fluid” describes a fluid generally
15 characterized as being a viscoelastic fluid comprising multiple components having inhomogeneous physical and/or chemical properties. It is the inhomogeneous properties of the multiple components that challenge the efficacy of an adsorbent material in the handling of complex fluids. In contrast with complex fluids, simple fluids, such as, for example, urine, physiological saline, water and the like, are generally characterized as
20 being relatively low-viscosity and comprising one or more components having homogeneous physical and/or chemical properties. As a result of having homogeneous properties, the one or more components of simple fluids behave substantially similarly during absorption or adsorption.

Although a complex fluid is generally characterized herein as including specific
25 components having inhomogeneous properties, each specific component of a complex fluid generally has homogeneous properties. Consider for example a hypothetical complex fluid having three specific components: red blood cells, blood protein molecules and water molecules. Upon examination, one skilled in the art could easily distinguish between each of the three specific components according to their generally
30 inhomogeneous properties. Moreover, when examining a particular specific component

such as the red blood cell component, one skilled in the art could easily recognize the generally homogeneous properties of the red blood cells.

The term "surface" and its plural generally refer herein to the outer or the topmost boundary of an object, material, structure, particle or the like.

5 As used herein, the phrase "absorbent article" refers to devices which absorb and contain body fluids, and more specifically, refers to devices which are placed against or near the skin to absorb and contain the various fluids discharged from the body. The term "disposable" is used herein to describe absorbent articles that are not intended to be laundered or otherwise restored or reused as an absorbent article after a single use.

10 Examples of such disposable absorbent articles include, but are not limited to: health care related products including surgical drapes, gowns, and sterile wraps; personal care absorbent products such as feminine hygiene products (*e.g.*, sanitary napkins, pantliners and the like), diapers, training pants, incontinence products and the like; as well as facial tissues.

15 Disposable absorbent articles such as, for example, many of the personal care absorbent products, typically comprise a fluid pervious topsheet, a liquid impervious backsheet joined to the topsheet and an absorbent core positioned between the topsheet and the backsheet. Disposable absorbent articles and components thereof, including the topsheet, backsheet, absorbent core and any individual layers of these components, have

20 a body-facing surface and a garment-facing surface. As used herein, "body-facing surface" means that surface of the article or component which is intended to be worn toward or placed adjacent to the body of the wearer, while the "garment-facing surface" is on the opposite side and is intended to be worn toward or placed adjacent to the wearer's undergarments when the disposable absorbent article is worn.

25 While a wide variety of adsorbent materials are known, the present invention relates, in one aspect, to the proper selection of adsorbent materials suitable for use in the handling of complex fluids such as, for example, blood, menses, loose passages, nasal discharges and the like. Adsorbent materials suitable for use in the handling of complex fluids desirably are substantially wettable or hydrophilic to complex fluids thus allowing

30 the complex fluids to spread over the surface of the adsorbent materials. In addition, it is desired that the adsorbent materials of the present invention are in particle form and

substantially insoluble in complex fluids. It is further desired that the adsorbent materials of the present invention are substantially inert and neither substantially soften nor substantially swell during adsorption. Any such suitable adsorbent material preferably has a high surface area relative to its weight as determined by a method such as gas
5 adsorption, cetyltrimethyl ammonium bromide adsorption or mercury intrusion porosimetry.

Adsorbent materials suitable for use in the present invention include, but are not limited to, organic materials, inorganic materials and mixtures thereof. Suitable inorganic materials include, for example, activated carbon, silicates, metal oxides,
10 zeolites, carbonates, phosphates, borates, aerogels and mixtures thereof. Suitable organic materials include, for example, cellulosic materials, starches, chitins, alginates, synthetic polymers and mixtures thereof.

The adsorbent material may optionally be treated with a surfactant or other surface-modifying agent prior to incorporation into any containment means. Many
15 materials are useful in this application, for example sulfonated alkyl and aryl compounds, ethoxylated alcohols and amines, polyamides and their derivatives, polysaccharides and their derivatives, polyethylene glycols and their derivatives, betaines and other zwitterionic compounds, and silyl compounds. Appropriate application methods are well known to one skilled in the art.

20 When used in a feminine hygiene product, the adsorbent material of the present invention will have a certain desirable distribution of pore sizes. In a bed of adsorbent particles, this will consist of the spaces between particles (interstitial spaces), as well as an internal pore structure of the particles themselves. These interstitial spaces are linked to form what can be considered as a network of interstitial spaces. When fluid moves
25 into or through a bed of particles, the fluid generally moves through these interstitial spaces. These interstitial spaces that the fluid moves through can also be referred to as interstitial pores.

Since the walls of an interstitial pore are the surfaces of the particles themselves, the shape and size of the interstitial pores are usually determined by the particles
30 themselves. Varying the size of the particles by varying their average dimensions or the distribution of their dimensions, varies the shape and size of the interstitial pores.

Interstitial pores play a significant role in the intake rate and retention of a complex fluid by adsorbent particles.

Adsorbent materials suitable for use desirably have an acceptable rate of intake with respect to complex fluids. This acceptable rate of intake can be achieved through a heterogeneous distribution of pore sizes. As previously discussed, a combination of particle sizes can provide an appropriate heterogeneous distribution of pore sizes. The pore size distribution may be measured with capillary tension, mercury porosimetry and, indirectly, by the permeability test, all of which are described herein. The inventors have found that pore sizes suitably range from about 1,000 to about 0.2 microns, with pore sizes between about 1,000 and about 100 microns primarily useful for the rapid intake and distribution of a complex fluid, and pore sizes between about 100 and about 0.2 microns primarily useful for the separation and retention of the components of a complex fluid.

Adsorbent particles are capable of retaining fluid in the interstitial pores or spaces between the particles as well as in the internal pores of the individual particles. It is desirable that the pores of an individual particle are accessible from the surface of the particle to adsorb the fluid. Fluid is capable of entering the internal pore volume of an individual particle through capillary forces. The addition of internal pores allows the fluid or liquid portion of the complex fluid to be retained by capillary force within the internal pores. This creates a dry feeling against the body and diminishes the amount of free liquid in the bed of adsorbent particles and, consequently, rewet. Suitable adsorbents have a range of internal pore sizes from about 100 to about 0.2 microns to adsorb different sized components of a complex fluid and thus minimize fluid rewet as measured by the Rewet and Centrifuge Retention test methods described herein.

If too many small pores are present, the liquid component of a complex fluid is removed too rapidly while the remainder of the complex fluid, mostly cells in the case of menses, does not spread. The inventors have determined it is desirable that the volume of pores less than 1 micron should be less than 2 percent of the total pore volume.

Based on the foregoing, adsorbent materials suitable for use in the present invention necessarily have at least the following parameters: wettable, stable to fluid

exposure, suitable interstitial pore size distribution for acceptable intake rate and suitable internal pore size distribution for desirable retention.

In various embodiments of the present invention other specific parameters of the adsorbent material may be desirable. For instance, when the complex fluid is menses and the adsorbent material is used in feminine hygiene products it is desired that the adsorbent materials of the present invention have a particle size of between about 1,000 to about 100; and, more desired, between about 850 to about 150 microns. The inventors have determined that particles of adsorbent material above about 1,000 microns are generally readily perceptible to the wearer of any containment means containing the adsorbent materials of the present invention, while particles of adsorbent material below 100 microns have been found to be difficult to contain within any containment means that would allow complex fluids to penetrate to the adsorbent materials. It is understood that the particles of adsorbent material falling within the range identified herein may comprise porous particles or may be agglomerated particles comprising many smaller particles of one or more adsorbent materials.

Another desirable specific parameter is capacity which is measured as the weight, in grams, of the complex fluid retained by the weight, in grams, of the adsorbent in the complex fluid retention capacity test method. For instance, when the complex fluid is menses and the adsorbent material is incorporated into a feminine hygiene product, it is desired that the complex fluid retention capacity of the adsorbent be between about 1 and about 15; alternatively, between about 2 and about 8; and finally, alternatively, between about 2 and about 6 g/g. It is believed that adsorbent materials having lower retention capacities than 2 g/g would require the use of such large amounts of adsorbent material that users may find the feminine hygiene product to be excessively heavy. The complex fluid retention capacity can be estimated by summing the amount of pore volume between about 100 and about 0.2 microns in diameter as determined by, for instance, capillary tension or mercury intrusion porosimetry. The complex fluid retention capacity is limited by the strength of the pore wall material.

As previously mentioned, a mixture of particle sizes is desirable to improve fluid intake and retention. Sufficient interstitial pores between particulates are needed so that menses can rapidly enter into the bed of adsorbent particles and be distributed between

the particulates. This property may be controlled with the particle size distribution of the adsorbent material. Generally, a broad particle size distribution is desired. A broad particle size distribution is used herein to describe a distribution having a standard deviation greater than 25 percent of the mean value.

5 If it is desired to move the fluid into and through the bed of particles relatively quickly, it is advantageous to minimize the variation in the interstitial pore size and shape along the length of the interstitial pore. Consequently, a relatively wide distribution of particle sizes would create interstitial pores that allow the fluid to move into and through the bed of particles relatively quickly. Should the variation in the size and the packing of
10 the particles become too large, such that some of the particles actually move within the interstitial pores themselves, the movement of fluid within and through the bed will not be relatively quick and may, instead, be relatively slow.

 The inventors have also found that a combination of pore sizes is effective at adsorbing a complex fluid. A bi-modal or multi-modal particle size distribution is
15 particularly desirable for producing a combination of pore sizes that is desirable for enhancing complex fluid intake and retention. One manner of achieving this desirable pore size distribution is to combine adsorbent particles of various sizes.

 Since the properties of interest for fluid intake are size and distribution of the pores, another manner of producing a pore size distribution suitable for the rapid intake
20 of a complex fluid consists of using relatively large, spherical particles. For instance, relatively large, spherical particles typically pack relatively poorly. This relatively poor packing of relatively large, spherical particles can lead to the existence of relatively large interstitial pores which allow a complex fluid to pass rapidly through a bed of relatively large, spherical particles. The Gel Bed Permeability Test Method described herein
25 measures the packing of particles in a bed. The inventors believe that permeability values above at least 1000 K for a bed of relatively large, spherical particles are indicative of relatively poor packing and consequently, a predictor of relatively rapid complex fluid intake.

 It should be noted that the present invention is not limited to the use of only one
30 of the adsorbent materials listed herein, but can also include mixtures of two or more adsorbent materials. As previously indicated, the adsorbent material is in particle form;

consequently, use of the phrase "adsorbent material" throughout the specification and appended claims includes a single particle of adsorbent material or an agglomeration of more than one particle of adsorbent material.

The adsorbent materials of the present invention may be appropriately contained
5 by a suitable containment means. Any means capable of containing the described adsorbent materials, which means is further capable of being located in a disposable absorbent article, is suitable for use in the present invention. Many such containment means are known to one skilled in the art. For example, the containment means may comprise a fibrous matrix such as an airlaid or wet laid web of cellulosic fibers, a
10 meltblown web of synthetic polymeric fibers, a spunbonded web of synthetic polymeric fibers, a coformed matrix comprising cellulosic fibers and fibers formed from a synthetic polymeric material, airlaid heat-fused webs of synthetic polymeric material, open-celled foams, and the like.

Alternatively, the containment means may comprise two layers of material which
15 are joined together to form at least one pocket or compartment containing adsorbent material. In such a case, at least one of the layers of material should be fluid-pervious. The second layer of material may be fluid-pervious or liquid-impervious. The layers of material may be cloth-like wovens and nonwovens, closed or open-celled foams, perforated films, elastomeric materials, or may be fibrous webs of material. When the
20 containment means comprises layers of material, the material should have a pore structure small enough or tortuous enough to contain the majority of adsorbent material. The containment means may also comprise a laminate of two layers of material between which the adsorbent material is located and contained.

Further, the containment means may comprise a support structure, such as a fiber
25 or a polymeric film, on which the adsorbent material is adhered. The adsorbent material may be adhered to one or both sides of the support structure which may be fluid-pervious or liquid-impervious.

The adsorbent material is present in the containment means in an amount of from
about 10 to about 100; alternatively, about 20 to about 100; alternatively, about 30 to
30 about 100; alternatively, about 40 to about 100; alternatively, about 50 to about 100; alternatively, about 60 to about 100; alternatively, about 70 to about 100; alternatively,

about 80 to about 100; and finally, alternatively, about 90 to about 100 weight percent based on total weight of the containment means and the adsorbent material.

In one specific embodiment of the present invention, the containment means comprises two layers of material which are joined to form a pocket adapted to contain the
5 adsorbent material. The two layers are suitably formed from any material capable of containing the adsorbent material including woven and nonwoven materials such as airlaid or wet laid fibers, meltblown fibers, spunbonded fibers, coformed fibers, binder fibers (such as bicomponent fibers), and the like, and are joined to form a pocket by heat fusion, sonic bonding, adhesives (such as water-soluble or water-sensitive adhesives,
10 latex adhesives, hot melt adhesives, or solvent-based adhesives) and the like. Clearly, a wide variety of materials may be employed to form the two layers and to join the two layers together to form the pocket. The adsorbent material is present in said pocket in an amount of from about 10 to about 100; alternatively, about 20 to about 100; alternatively, about 30 to about 100; alternatively, about 40 to about 100; alternatively, about 50 to
15 about 100; alternatively, about 60 to about 100; alternatively, about 70 to about 100 weight percent; alternatively, about 80 to about 100 weight percent; and finally, alternatively, about 90 to about 100 weight percent based on total weight of the adsorbent material present in the pocket and the weight of the two layers forming the pocket. In addition to the adsorbent material, the pocket may contain a fibrous material or other
20 filler material that does not unacceptably affect the adsorbent properties of the adsorbent material.

In another embodiment, the containment means comprises a matrix of fibers. The adsorbent material is mixed with the fibers of the matrix. The adsorbent material is present in the mixture of fibers and adsorbent material in an amount of from about 20 to
25 about 95; alternatively, about 30 to about 85 weight percent; and finally, alternatively, about 50 to about 75 weight percent based on total mixture weight.

Any fibers capable of containing an adsorbent material and of forming a composite when in combination with the adsorbent material are believed suitable for use in the present invention. It is often preferred that the fibers are hydrophilic. As used
30 herein, a fiber will be considered to be "hydrophilic" when it possesses a contact angle of water in air of less than 90 degrees. For the purposes of this application, contact angle

measurements are determined as set forth by Good and Stromberg in "Surface and Colloid Science" Vol. 11 (Plenum Press, 1979).

Fibers suitable for use in the present invention include cellulosic fibers such as wood pulp fluff, cotton, cotton linters, rayon, cellulose acetate, and the like, as well as synthetic polymeric fibers. The synthetic polymeric fibers may be formed from inherently hydrophilic polymeric materials or may be formed from inherently hydrophobic polymeric materials (water in air contact angle of greater than 90 degrees), which fibers are then treated to render at least the outer surface of the fibers hydrophilic. For example, hydrophilic fibers may be formed from an intrinsically hydrophilic polymer such as a block copolymer of nylon, e.g., nylon-6, and a polyethylene oxide diamine. Such block copolymers are commercially available from Allied-Signal Inc. under the trade designation HYDROFIL. Alternatively, the fibers may be formed from an intrinsically hydrophobic polymer such as a polyolefin or polyester which has been surface modified to provide a generally nonfugitive hydrophilic surface. Such surface modified polyethylene is commercially available from the Dow Chemical Company under the trade designation ASPUN wettable polyethylene.

When the hydrophilic fibers are formed by applying a hydrophilic surface treatment to a generally hydrophobic polymer, it is believed desirable to employ a generally non-fugitive surface treatment in order to obtain the desired performance.

The synthetic polymeric fibers suitable for use in the present invention may be formed by a melt-extrusion process wherein fibers of a polymeric material are extruded and attenuated to produce fibers having a desired diameter. Alternatively, the fibers may be formed through a spinning process. Any fiber-producing process known to one skilled in the art is believed to be suitable for use in the present invention.

Fibers suitable for use in the present invention generally have a length of at least about 1 millimeter. The fibers may have a maximum length approaching infinity. That is to say, the fibers may be essentially continuous such as those fibers formed through a meltblowing process under certain conditions known to one skilled in the art.

Reference to a mixture of fibers and adsorbent material is intended to refer to a situation in which the adsorbent material is in direct contact with the fibers or is not substantially prevented from migrating into contact with the fibers. Thus, for example,

in a multi-layered adsorbent core in which the first layer comprises an airlaid mixture of wood pulp fluff and adsorbent material and the second layer comprises only airlaid fluff, only the first layer is considered a mixture of fibers and adsorbent material provided, however, substantial dry migration of the adsorbent material between the two layers is prevented. Methods of preventing such migration are known and include separating the layers by a tissue wrap sheet, high density fiber layer or similar means to prevent substantial dry migration of the adsorbent material between the two layers. The mixture of adsorbent material and fibers may be relatively homogenous or relatively non-homogeneous. In the case of a non-homogeneous mixture, the adsorbent material may be arranged in a gradient or may be layered with the fibers.

When the containment means comprises a mixture of fibers and adsorbent material, the mixture of fibers and adsorbent material may be formed in a variety of ways. For example, the mixture may be formed by airlaying or wetlaying the fibers and adsorbent material, according to processes known in the art, to form batts of the mixture. Airlaying the mixture of fibers and adsorbent material is intended to encompass both the situation wherein preformed fibers are airlaid with the adsorbent material as well as the situation in which the adsorbent material is mixed with the fibers as the fibers are being formed, such as through a meltblowing process.

The adsorbent materials of the present invention are particularly suitable for use in disposable absorbent articles. In general, the adsorbent materials may be used in the same manner for which conventional odor-controlling adsorbent materials have been used, for example, in laminates, in relatively high density cores (*i.e.*, compacted cores, calendered cores, densified cores, etc.), or in relatively low density cores (*i.e.*, not compacted, for example, air-laid cores). However, the adsorbent materials of the present invention provide certain advantages over conventional odor-controlling adsorbent materials. In general, when compared to conventional adsorbent materials, the adsorbent materials of the present invention demonstrate an improved efficacy in the handling of complex fluids. In particular, the adsorbent materials of the present invention demonstrate an improved efficacy in the handling of menses. As a result of this improved efficacy, the adsorbent materials of the present invention provide product developers with the flexibility either to complement absorbent materials typically

contained in disposable absorbent articles with the addition of sufficient quantities of the adsorbent material of the present invention or to replace certain absorbent materials typically contained in disposable absorbent articles with sufficient quantities of the adsorbent material of the present invention.

5

Test Methods

Intake Rate and Rewet Test Method

As used herein, the Intake Rate and Rewet Test Method measures at least the
10 following two characteristics of materials:

1. Intake rate - the amount of time, in seconds, it takes for a known amount of material to intake multiple insults of known quantities of a fluid; and
2. Rewet - the amount of fluid, in grams, that is released from the material when blotter paper is placed on top of the material and a known pressure is applied
15 for a predetermined period of time.

Testing according to this method consisted of using a stopwatch to determine the amount of time, in seconds, required for 20 mL of material to intake multiple insults (1 or 2 mL) of fluid. A Harvard Syringe Pump is programmed to dispense 2 mL of fluid onto 20 mL of absorbent material, at which time a stopwatch is simultaneously started.
20 The stopwatch is stopped when the 2 mL of fluid is taken into the material. A second insult of 2 mL is then dispensed and timed. The second insult is followed by a third insult, this time consisting of 1 mL, which is also timed. This results in a total of 5 mL and three timed insults. Wait approximately 60 seconds from intake of the third insult before placing a pre-weighed blotter paper onto the 20 mL of material and applying a 0.5
25 psi pressure for 60 seconds. After 60 seconds, the blotter paper is reweighed and the fluid, in grams, that has been absorbed by the blotter paper is considered the amount of rewet. Testing is typically conducted under TAPPI Standard Conditions.

Equipment and Materials:

- Harvard Apparatus Programmable Syringe Pump, Model No. 44, commercially available from Harvard Apparatus, South Natick, MA 01760 USA.
- 5 • The fluid in this instance, by way of example only and not by way of limitation, is an artificial menses (simulant), disclosed in U.S. Patent No. 5,883,231, issued March 16, 1999, to Achter et al., the disclosure of which is hereby incorporated herein by reference to the extent that said disclosure is consistent (*i.e.*, not contradictory) with the present specification. The
10 simulant disclosed and claimed in U.S. Patent No. 5,883,231 is commercially available from Cocalico Biologicals, Inc., 449 Stevens Rd., P.O. Box 265, Reamstown, PA 17567 USA.
- Disposable plastic weighing boats commercially available from NCL of Wisconsin, Inc., Birnamwood, WI 54414 USA, part number W-D 80055.
- 15 • 60 cc disposable syringe, commercially available from Becton Dickinson, Franklin Lakes, NJ 07417 USA; Tygon tubing, size 16 with 0.12" inner diameter, part number 6409-16, commercially available from Cole-Parmer Instrument Company, Chicago, IL 60648 USA; and 1/8" outer diameter hose, barb size, part number R-3603 and also commercially available from Cole-
20 Parmer Instrument Company.
- 5.5 cm blotter paper, commercially available from VWR Scientific Products, 1145 Conwell Ave., Willard, OH 44890 USA, catalogue number 28310-015.
- Weight, made by taking a 100 mL Pyrex beaker and filling it with any suitable substance to 717.5 grams to obtain a 0.5 psi loading.
- 25 • Balance, readable to 0.001 g (Note: standards should be NIST traceable and should be recertified at a frequency adequate to assure accuracy).
- Stopwatch, readable to 0.1 s (Note: stopwatch should be NIST traceable).
- Graduated cylinder readable to 20 mL.
- Clear acrylic plate (of a size sufficient to be supported on top of a disposable
30 plastic weighing boat) with a hole drilled approximately in the center thereof for insertion of the Tygon tubing.

Specimen Preparation:

The simulant is removed from a refrigeration unit, placed on a rotator and then gently rotated for approximately 30 minutes to thoroughly mix the contents and bring the simulant to room temperature.

The graduated cylinder is placed onto the balance and the weight tared. 20 mL of material is introduced into the graduated cylinder. The graduated cylinder is removed from the balance. The bottom of the graduated cylinder is gently tapped on the top of the lab bench or similar hardened surface approximately 10 times to induce settling. Visual inspection is made to ensure that there is 20 mL of material in the graduated cylinder. The 20 mL of material is poured into a weighing boat and the material is gently leveled.

The Harvard Syringe Pump is set to the Program Mode. The Infuse Rate is set to 12 mL/min. with the Target Volume set to 2 mL. Diameter is set to the correct syringe size. The Harvard Syringe Pump is filled with approximately 60 mL of simulant.

The steps of the testing method are as follows:

1. One end of the Tygon tubing is inserted through the hole in the acrylic plate.
2. The acrylic plate is placed over a weighing boat containing 20 mL of absorbent material. The Tygon tubing should be placed approximately over the center of the material.
3. Simultaneously start the stopwatch and begin dispensing the first 2 mL insult of simulant.
4. Stop the stopwatch when the simulant is taken in by the material. The reading on the stopwatch is recorded as "Insult 1" in seconds. In the event that the simulant is not taken in by the material being tested (*i.e.*, the simulant sits on the top of the material) within five minutes, stop the test and record 300+ seconds.
5. Simultaneously start the stopwatch and begin dispensing the second 2 mL insult of simulant.
6. Stop the stopwatch when the simulant is taken in by the material. The reading on the stopwatch is recorded as "Insult 2" in seconds. In the event that the simulant is not taken in by the material being tested (*i.e.*, the simulant sits on

the top of the material) within five minutes, stop the test and record 300+ seconds.

7. Simultaneously start the stopwatch and begin dispensing the simulant. In this instance, however, the Harvard Syringe Pump is halted after 1 mL of simulant has been dispensed.
8. Stop the stopwatch when the 1 mL of simulant is taken in by the material. The reading on the stop watch is recorded as "Insult 3" in seconds. Once again, should the simulant not be taken in by the material being tested (*i.e.*, the simulant sits on the top of the material) within five minutes, stop the test and record 300+ seconds.
9. Wait 60 seconds after the third insult is taken in by the material.
10. Weigh two pieces of blotter paper and record this weight as "BP Dry."
11. At the end of the 60 seconds noted in step 9, gently place the blotter paper on the material and then gently place the 0.5 psi weight onto the blotter paper and start the stopwatch.
12. After 60 seconds, remove the weight and reweigh the blotter paper. This weight of the blotter paper is recorded as "BP Wet."

Steps 3 through 12 outlined above are repeated until the simulant is no longer taken in by the material (*i.e.*, the simulant sits on the top of the material and is not taken in within five minutes).

The results of the rewet portion of the test method are recorded in grams and calculated as follows:

$$(\text{BP Wet}) - (\text{BP Dry}) = \text{Rewet}$$

25

Method for Determining Retention Capacity

As used herein, the Method for Determining Retention Capacity measures the amount of test fluid that a sample of material retains after a centrifugal force has been applied. The amount of fluid retained is calculated as a gram per gram retention. The test is typically conducted under TAPPI Standard Conditions. When the test fluid is a complex fluid, such as, for example, blood, menses, artificial menses (simulant), loose

passages, nasal discharges and the like, the retention capacity of a material is sometimes referred to as a complex fluid retention capacity (CFRC).

In general, testing according to this method is performed by placing a 0.5 g sample of material into a modified cylinder, exposing the sample of material to a desired fluid for 60 minutes and then placing the cylinders into a centrifuge to remove excess fluid. The results are calculated to obtain the grams of fluid retained per gram of sample of material.

Equipment and Materials:

- 10 • Artificial menses fluid (simulant), disclosed in U.S. Patent No. 5,883,231, issued March 16, 1999, to Achter et al. The simulant disclosed and claimed in U.S. Patent No. 5,883,231 is commercially available from Cocalico Biologicals, Inc. 449 Stevens Rd., P.O. Box 265, Reamstown, PA 17567 USA.
- 15 • Sorvall RT 6000D centrifuge, commercially available from Global Medical Instrumentation, Inc., 3874 Bridgewater Dr., St. Paul, MN 55123 USA.
- Four 200 mL, screw top centrifuge bottles, commercially available from International Equipment Co., 300 Second Ave., Needham Heights, MA 02494 USA.
- 20 • Balance, readable to 0.001 g (Note: standards should be NIST traceable and should be recertified at a frequency adequate to assure accuracy).
- Four 50 mL Pyrex beakers.
- Lab timer, 60 minute capacity, readable to one second, commercially available from VWR Scientific Products, 1145 Conwell Ave., Willard, OH 44890 USA.
- 25 • Four modified Lexan cylinders, 9 cm high, 3.1 cm ID, 4.8 cm OD, with a 300 holes/in² screen attached to the bottom.
- U.S. standard 30 and 50 screen sieves, 8 inch diameter, 2 inch height, commercially available from VWR Scientific Products, 1145 Conwell Ave., Willard, OH 44890 USA, catalogue numbers 57334-456 and 57334-464, respectively.
- 30

- Stainless steel screen, 4 holes per inch or enough open space to allow simulant to drain.

Specimen Preparation:

5 Prepare the sample of material by using the U.S. standard 30 and 50 screen sieves to fractionate a sample to the 300 to 600 micron size. Store the fractionated sample of material in a sealed substantially airtight container for use when the sample or samples of material will be prepared. The modified cylinder is placed on the balance and the weight tared. Place 0.5 g \pm 0.005 g of the fractionated sample into one of the modified
10 cylinders. Record this weight as Sample Weight. The modified cylinder containing the sample of material is weighed and this weight is recorded as Dry Cylinder Weight. Additional samples of material are placed in the three remaining modified cylinders according to the foregoing steps.

The simulant is removed from a refrigeration unit, placed on a rotator and then
15 gently rotated for approximately 30 minutes to thoroughly mix the contents and bring the simulant to room temperature.

The steps of the testing method are as follows:

1. Approximately 10 mL of simulant are placed into a 50 mL Pyrex beaker.
2. A modified cylinder containing the sample of material is placed into the 50
20 mL Pyrex beaker.
3. Approximately 15 mL of simulant are poured into the modified cylinder. This ensures that the sample of material has access to the simulant from both above and below.
4. Repeat step 1 through 3 as necessary for any desired additional sample of
25 material.
5. After step 4 has been completed, the timer is set for 60 minutes and started.
6. After 60 minutes have elapsed, the modified cylinders are removed from the Pyrex beakers and placed on the stainless steel screen for 60 seconds.
7. After 60 seconds, the modified cylinders are removed from the stainless steel
30 screen and placed in the 200 mL centrifuge bottles.
8. The centrifuge bottles are placed in the centrifuge for 3 minutes at 1,200 rpm.

9. After 3 minutes, the modified cylinders are removed from the centrifuge bottles and the modified cylinders containing the samples of material are weighed. This weight is recorded as Wet Cylinder Weight.
- 5 The Complex Fluid Retention Capacity ("CFRC") of each sample of absorbent is then calculated according to the following formula:

$$\frac{[(\text{Wet Cylinder Weight} - \text{Dry Cylinder Weight}) - \text{Product Weight}]}{(\text{Product Weight})}$$

10

Where reported in any of the following examples, the Retention Capacities are an average of two samples (*i.e.*, $n = 2$).

Capillary Tension Test Method

- 15 The Capillary Tension Test (CTT) is a test which measures the ability of an absorbent material to absorb a liquid (0.9 weight percent solution of sodium chloride in distilled water) while under an applied load or restraining force while subjected to a negative pressure gradient.

Referring to FIG. 1, the apparatus and method for determining CTT values will be described. Shown is a perspective view of the apparatus in position during a test.

- Shown is a laboratory stand (31) which is graduated in centimeters and which has an adjustable collar (32) for raising and lowering a support ring (33). The support ring (33) supports a funnel (34) which has a 6 centimeter diameter. In the funnel (34) is placed a porous glass plate (35) which has a nominal maximum pore diameter of about 40 to about 60 micrometers. To the bottom of the funnel (34) is attached a first flexible plastic tubing (36) which is attached at its other end to a rigid plastic tube (37) which is held in place with a clamp (38). The other end of the rigid plastic tube (37) is attached to a second flexible plastic tubing (39) which is attached at its other end to a vented liquid reservoir (40). The vented liquid reservoir (40) rests on a balance (41) which is attached to a recorder (42) which is used to record the weight loss of liquid from the vented liquid reservoir (40) as liquid is absorbed by the sample being evaluated.
- 20
- 25
- 30

A plastic sample cup (43), which contains the material sample (44) to be tested, has a liquid-permeable bottom and rests on top of the porous glass plate (35) in the funnel (34). A weight (46) rests on top of a spacer disc (45) resting on top of the material sample.

5 The sample cup (43) consists of a plastic cylinder having a 1 inch inside diameter and an outside diameter of 1.25 inches. The bottom of the sample cup (43) is formed by adhering a 100 mesh metal screen having 150 micron openings to the end of the cylinder by heating the screen above the melting point of the plastic and pressing the plastic cylinder against the hot screen to melt the plastic and bond the screen to the plastic
10 cylinder.

To carry out the test, a volume of 10.12 cubic centimeters of sample material (44) is placed into the sample cup (43). A 100 gram weight (46) is then placed on top of the spacer disc, thereby applying a load of about 0.3 pound per square inch. The sample cup is placed on the porous glass plate (35).

15 A negative pressure gradient is established by lowering the funnel (34) until liquid from the vented liquid reservoir (40) flows through the tubing (37, 38, and 39) into the funnel (34) to make contact with the porous glass plate (35). The funnel (34) is then raised to a level along the graduated laboratory stand (31) until a desired negative pressure gradient (as measured in difference in height, in centimeters, between the top of the
20 liquid level in the vented liquid reservoir (40) and the level of the porous glass plate (35)) is established. The recorder is then allowed to measure the amount of liquid which is removed from the vented liquid reservoir (40) and absorbed by the absorbent material, on a gram of liquid absorbed per gram of material basis, as a function of the negative pressure gradient. The negative pressure gradient is inversely proportional to the
25 equivalent radius of the interstitial pores according to the following equation:

$$R = (2\gamma\cos\theta)/(\delta gh)$$

where R = equivalent radius of interstitial pores, γ = surface tension of the fluid, θ = receding contact angle, δ = fluid density, g = acceleration of gravity, and h = negative pressure gradient.

30 From this test method, a table of cumulative pore size volume as a function of the equivalent radius of the interstitial pore can be generated.

Gel Bed Permeability Test Method

A suitable piston/cylinder apparatus for performing the Gel Bed Permeability (GBP) test is shown in FIGs. 2 and 3. Referring to FIG. 2, an apparatus (120) consists of a cylinder (122) and a piston (generally indicated as 124). As shown in FIG. 2, the piston (124) consists of a cylindrical LEXAN shaft (126) having a concentric cylindrical hole (128) bored down the longitudinal axis of the shaft. Both ends of the shaft (126) are machined to provide first and second ends (130, 132). A weight (134) rests on the first end (130) and has a cylindrical hole (136) bored through the center thereof. Inserted on the second end (132) is a circular piston head (140). The piston head (140) is sized so as to vertically move inside the cylinder (122). As shown in FIG. 3, the piston head (140) is provided with inner and outer concentric rings containing seven and fourteen approximately 0.375 inch (0.95 cm) cylindrical holes, respectively (indicated generally by arrows 142 and 144). The holes in each of these concentric rings are bored from the top to bottom of the piston head (140). The piston head (140) also has a cylindrical hole (146) bored in the center thereof to receive the second end (132) of the shaft (126).

Attached to the bottom end of the cylinder (122) is a No. 400 mesh stainless steel cloth screen (148) that is biaxially stretched to tautness prior to attachment. Attached to the bottom end of the piston head (140) is a No. 400 mesh stainless steel cloth screen (150) that is biaxially stretched to tautness prior to attachment. A sample of adsorbent material (152) is supported on the screen (148).

The cylinder (122) is bored from a transparent LEXAN rod or equivalent and has an inner diameter of 6.00 cm (area = 28.27 cm²), a wall thickness of approximately 0.5 cm, and a height of approximately 5.0 cm. The piston head (140) is machined from a LEXAN rod. It has a height of approximately 0.625 inches (1.59 cm) and a diameter sized such that it fits within the cylinder (122) with minimum wall clearances, but still slides freely. A hole (146) in the center of the piston head (140) has a threaded 0.625 inch (1.59 cm) opening (18 threads/inch) for the second end (132) of the shaft (126). The shaft (126) is machined from a LEXAN rod and has an outer diameter of 0.875 inches (2.22 cm) and an inner diameter of 0.250 inches (0.64 cm). The second end (132) is approximately 0.5 inches (1.27 cm) long and is threaded to match the hole (146) in the piston head (140). The first end (130) is approximately 1 inch (2.54 cm) long and 0.625

inches (1.58 cm) in diameter, forming an annular shoulder to support the stainless steel weight (134). The annular stainless steel weight (134) has an inner diameter of 0.625 inches (1.59 cm), so that it slips onto the first end (130) of the shaft (126) and rests on the annular shoulder formed therein. The combined weight of the piston (124) and the weight (134) equals approximately 596 g, which corresponds to a pressure of 0.30 psi (20,685 dynes/cm²), for an area of 28.27 cm².

When fluids flow through the piston/cylinder apparatus, the cylinder (122) generally rests on a 16-mesh, rigid stainless-steel support screen (not shown) or equivalent. The piston and weight are placed in an empty cylinder to obtain a measurement from the bottom of the weight to the top of the cylinder. This measurement is taken using a caliper readable to 0.01 mm. This measurement will later be used to calculate the height of the bed of the sample of adsorbent material (152). It is important to measure each cylinder empty and keep track of which piston and weight were used. The same piston and weight should be used for measurement when the sample of adsorbent material is swollen.

The adsorbent layer used for GBP measurements is formed by swelling approximately 0.9 g of a sample of adsorbent material in the GBP cylinder apparatus (dry adsorbent material should be spread evenly over the screen of the cylinder prior to swelling) with a fluid, typically 0.9% (w/v) aqueous NaCl, for a time period of approximately 15 minutes. The sample of adsorbent material is taken from a population of adsorbent material that is prescreened through US standard 30 mesh and retained on US standard 50 mesh. The adsorbent material, therefore, has a particle size of between 300 and 600 microns. The particles may be prescreened by hand or automatically prescreened with, for example, a Ro-Tap Mechanical Sieve Shaker Model B, commercially available from W. S. Tyler, Inc., Mentor, OH USA.

At the end of the 15 minute period, the cylinder is removed from the fluid and the piston/weight assembly is placed on the sample of adsorbent material. The thickness of the swollen sample of adsorbent material is determined by measuring from the bottom of the weight to top of the cylinder with a micrometer. The value obtained when taking this measurement with the empty cylinder is subtracted from the value obtained after swelling

the sample of adsorbent material. The resulting value is the height of the bed of the swollen sample of adsorbent material, H.

The GBP measurement is initiated by adding the fluid to the cylinder (122) until the fluid attains a height of 4.0 cm above the bottom of the sample of adsorbent material (152). This fluid height is maintained throughout the test. The quantity of fluid passing through the sample of adsorbent material (152) versus time is measured gravimetrically. Data points are collected every second for the first two minutes of the test and every two seconds for the remainder. When the data are plotted as quantity of fluid passing through the bed of the sample of adsorbent material versus time, it becomes clear to one skilled in the art when a steady flow rate has been attained. Only data collected once the flow rate has become steady is used in the flow rate calculation. The flow rate, Q, through the sample of adsorbent material (152), is determined in units of g/s by a linear least-square fit of fluid passing through the sample of adsorbent material (in grams) versus time (in seconds).

Permeability in cm^2 is obtained by the following equation:

$$K = [Q \cdot (H \cdot \mu)] / [A \cdot \rho \cdot P]$$

Where K = Gel Bed Permeability (cm^2); Q = flow rate (g/sec); H = height of bed of sample of adsorbent material (cm); μ = liquid viscosity (poise); A = cross-sectional area for liquid flow (cm^2); ρ = liquid density (g/cm^3); and P = hydrostatic pressure (dynes/cm^2) (normally approximately 3,923 dynes/cm^2).

EXAMPLES

The following Examples describe various embodiments of the invention. Other embodiments within the scope of the claims herein will be apparent to one skilled in the art from consideration of the specification or practice of the invention as disclosed herein. It is intended that the specification, together with the Examples, be considered exemplary only, with the scope and spirit of the invention being indicated by the claims which follow the Examples.

Example 1

This Example demonstrates that a silica adsorbent is well suited for handling complex fluids such as menses. The adsorbent material utilized in this Example was

Zeofree 5175B, a granulated adsorbent material commercially available from J.M. Huber Corp., Havre de Grace, MD USA. Granulation is achieved physically and no ingredients are present other than the precipitated silica particles. Zeofree 5175B as received was evaluated for intake rate and rewet. The results of this evaluation are provided in TABLE

5 1.

TABLE 1

	Zeofree 5175B
Insult 1 (s)	45.91
Insult 2 (s)	62.34
Insult 3 (s)	63.0
Rewet (g)	0.85

The complex fluid retention capacity (using simulant) for the foregoing sample was at least about 2.05 g/g.

10

Example 2

This Example illustrates the effect of particle size and distribution on intake of Zeofree 5175B, a precipitated silica adsorbent material commercially available from J.M. Huber Corp., Havre de Grace, MD USA. In this Example, the Zeofree 5175B was sieved
15 into a finely graded series of particle size distributions. These distributions were evaluated for intake rate and rewet "as is" and also in combinations to illustrate that intake rate can be effected through an appropriate combination of particles having varying sizes. The results of these evaluations are provided in TABLE 2a and illustrate that a narrow particle size distribution is not effective at adsorbing the fluid relatively quickly.

TABLE 2a

Mesh	Insult 1 (s)	Insult 2 (s)	Insult 3 (s)	Rewet (g)
As received	45.91	62.34	63.0	0.85
>20	300+	---	---	---
20-30	300+	---	---	---
30-40	300+	---	---	---
40-50	236	300+	---	---
50-60	300+	---	---	---
60-70	300+	---	---	---
70-80	268	300+	---	---
80-100	300+	---	---	---
100-140	300+	---	---	---
140-170	300+	---	---	---
30-50	178.0	122	184	0.74

TABLE 2b below illustrates that when narrow particle size distributions are recombined into bimodal particle size distributions, some combinations can intake fluid with intake times of less than 150 seconds, which is considered desirable. Suitable combinations contain a larger percentage of coarse materials. Adequate ratios from fine to coarse include, desirably 10/90; more desirably, 20/80; and, most desirably, 25/75 mesh. These recombined ratios can be characterized as having a standard deviation greater than 25 percent of the mean.

TABLE 2b

50:30 Screen Ratio (%)	Insult 1 (s)	Insult 2 (s)	Insult 3 (s)
10/90	92	110	145
20/80	67	124	134.5
25/75	60	107	169
50/50	209.5	260	140
75/25	216	300+	---
80/20	206	146	---
90/10	160	168	185

TABLE 2c illustrates interstitial pore size distribution for sieved Zeofree 5175B between about 30 and about 50 mesh screen as determined by the Capillary Tension Test Method. TABLE 2d illustrates interstitial pore size distribution for Zeofree 5175B having a ratio of 75% of between about 20 to about 30 mesh material and 25% of between about 40 to about 50 mesh material as determined by the Capillary Tension Test Method. The pore size distribution produced by the combination of the two sets of particle sizes shown in TABLE 2d contained a smaller percentage of small pores than the sample shown in TABLE 2c. The relatively greater percentage of pores larger than about 100 microns for the combined particle mixture correlates with an improvement of intake as illustrated by the data in TABLES 2a and 2b.

TABLE 2c

Pore Radius (microns)	Cumulative Pore Volume (g/g)	% Void Volume (cumulative)
50.00	2.27	76.96
100.00	2.30	77.94
150.00	2.39	81.15
200.00	2.49	84.56
250.00	2.59	87.86
300.00	2.70	91.52
350.00	2.78	94.14
400.00	2.79	94.75
450.00	2.81	95.20
500.00	2.82	95.49
1000.00	2.95	100.00

TABLE 2d

Pore Radius (microns)	Cumulative Pore Volume (g/g)	% Void Volume (cumulative)
50.00	2.08	70.24
100.00	2.08	70.34
150.00	2.09	70.74
200.00	2.15	72.67
250.00	2.35	79.28
300.00	2.49	84.14
350.00	2.59	87.71
400.00	2.67	90.39
450.00	2.73	92.27
500.00	2.75	93.09
1000.00	2.96	100.00

5

Example 3

This Example illustrates that Vermiculite, a hydrated laminar magnesium-aluminum-iron-silicate (commercially available from Strong-Lite Products, P.O. Box 8029, Pine Bluff, AR 71611 USA), exhibits desirable intake time, rewet and complex fluid retention capacity. TABLE 3b illustrates that Vermiculite exhibits suitable pore size

distribution to provide for desirable intake and rewet. The pore size distribution from TABLE 3b has been determined by the Capillary Tension Test Method. It is believed that the laminar internal pore structure of Vermiculite contributes to this adsorbent material exhibiting desirable intake, rewet and complex fluid retention capacity.

5

TABLE 3a

	Vermiculite
Insult 1 (s)	29.03
Insult 2 (s)	30.56
Insult 3 (s)	19.62
Rewet (g)	0.32

TABLE 3b

Pore Radius (microns)	Cumulative Pore Volume (g/g)	% Void Volume (cumulative)
50.00	2.87	57.93
100.00	2.94	59.25
150.00	3.13	63.10
200.00	3.55	71.65
250.00	3.95	79.58
300.00	4.23	85.26
350.00	4.37	88.09
400.00	4.54	91.62
450.00	4.61	93.00
500.00	4.67	94.13
1000.00	4.96	100.00

Vermiculite exhibited a complex fluid retention capacity of at least about 2.17 g/g.

5

Example 4

This Example illustrates that Ryolex 3, primarily an aluminum silicate adsorbent material (commercially available from Silbrico Corp., 6300 River Rd., Hodgkins, IL 60525-4257 USA) known as perlite, exhibits desirable intake time as illustrated in TABLE 4a. TABLE 4b illustrates that Ryolex 3 exhibits suitable pore size distribution to provide
 10 for desirable intake. The pore size distribution from TABLE 4b has been determined by the capillary tension test method. Ryolex 3 exhibited a complex fluid retention capacity of at least about 4.4 g/g.

TABLE 4a

	Ryolex 3
Insult 1 (s)	42.45
Insult 2 (s)	59.88
Insult 3 (s)	80.27
Rewet (g)	1.08

TABLE 4b

Pore Radius (microns)	Cumulative Pore Volume (g/g)	% Void Volume (cumulative)
50.00	3.06	72.36
100.00	3.26	77.20
150.00	3.45	81.60
200.00	3.58	84.79
250.00	3.70	87.64
300.00	3.76	89.00
350.00	3.82	90.49
400.00	3.88	91.75
450.00	3.91	92.48
500.00	3.93	92.97
1000.00	4.23	100.00

Example 5

This Example illustrates that Celphere CP305, a spherulated microcrystalline
 5 cellulose (commercially available from FMC Corp., 1735 Market St., Philadelphia, PA
 19103 USA) exhibits desirable intake, and rewet as illustrated in TABLE 5.

TABLE 5

	Celphere CP305
Insult 1 (s)	31.89
Insult 2 (s)	56.99
Insult 3 (s)	47.78
Rewet (g)	0.00

Celphere CP305 is an example of a relatively spherical adsorbent material that displays a
 10 uniform particle size distribution (*i.e.*, the average particle size diameter is about 400
 microns). Since the particles are rather large and relatively spherical, the permeability of
 this material is high (*i.e.*, above about 1,000 K). This high permeability explains the
 desired intake. However, since Celphere CP305 has no internal structure, the complex

fluid retention capacity was about 0.63 g/g and illustrates the need for pores less than about 100 microns for retention of a complex fluid.

Example 6

- 5 This Example illustrates that LC200 HF, a material made of pelletized cellulose fibers (commercially available from J. Rettenmaier & Söhne GmbH & Co., D-73496, Rosenberg, Germany) exhibits desirable intake and rewet as illustrated in TABLE 6.

TABLE 6

	LC200 HF
Insult 1 (s)	34
Insult 2 (s)	38
Insult 3 (s)	26
Rewet	0.75

10

Example 7

- Cab-O-Sil M5, an untreated fumed silica (commercially available from Cabot Corp. Boston, MA 02109 USA)), illustrates the effect on intake of a complex fluid (simulant in this instance) caused by fluid de-watering due to the presence of too high a percentage (>2 percent) of small pores (<1 micron). TABLE 7a illustrates the effect of an excessive
- 15 volume of pores sizes less than about 1 micron on the intake rate for a complex fluid.

TABLE 7a

	Cab-O-Sil M5
Insult 1 (s)	300+
Insult 2 (s)	---
Insult 3 (s)	---

TABLE 7b

Pore Diameter (microns)	Cumulative Pore Volume (g/g)	% Pore Volume (cumulative)
653.512	12.6411	100.0
378.860	12.4288	98.3
259.830	12.002	94.9
200.326	11.4777	90.8
162.243	11.0606	87.5
135.774	10.6848	84.5
117.322	10.3533	81.9
102.620	9.9384	78.6
90.892	9.6361	76.2
81.925	9.3729	74.1
74.171	9.1418	72.3
68.064	8.808	69.7
62.602	8.58	67.9
58.144	8.3835	66.3
54.149	8.0819	63.9
50.265	7.8929	62.4
47.840	7.7631	61.4
45.261	7.6236	60.3
39.375	7.2523	57.4
30.661	6.6537	52.6
20.600	5.7078	45.2
10.037	4.0636	32.1
8.037	3.621	28.6
6.030	3.0944	24.5
4.101	2.5759	20.4
2.007	1.7004	13.5
1.039	1.0323	8.2
0.512	0.4653	3.7
0.251	0	0.0

The data of TABLE 7b illustrate that the pore size distribution for Cab-O-Sil, determined by mercury porosimetry, contains about 8 percent of the pore volume in pores of less than about 1 micron in diameter. Mercury porosimetry data was obtained from Micromeritics Instrument Corp., One Micromeritics Dr., Norcross GA 30093 USA. The test requested was Macro and Meso Volume/Size Distribution by Mercury Intrusion Porosimetry, Test No. 005-65000-31. The sample was run on Micromeritics Instrument

Corp.'s AutoPore Mercury Porosimeter, Unit 750, beginning on July 28, 1999, at 17:00 hours and concluding on July 29, 1999, at 10:00 hours.

5 The above-disclosed Examples are not intended to limit the scope of the present invention in any way. Various modifications and other embodiments and uses of the disclosed superabsorbent-containing composites, apparent to those of ordinary skill in the art, are also considered to be within the scope of the present invention.

What is claimed is:

1. An absorbent article comprising a containment means and particles of at least one adsorbent material, while in the containment means about 20 to about 50 percent of the adsorbent particles have a pore volume coming from pores greater than about 100 microns for complex fluid intake and complex fluid distribution, and about 80
5 to about 50 percent of the pore volume coming from pores smaller than about 100 microns for complex fluid retention, the adsorbent particles being present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.
2. The absorbent article of claim 1, wherein the containment means comprises a topsheet and a backsheet.
3. The absorbent article of claim 2, wherein the topsheet is a fluid-pervious nonwoven.
4. The absorbent article of claim 2, wherein the topsheet is an apertured film.
5. The absorbent article of claim 2, wherein the backsheet is a nonwoven.
6. The absorbent article of claim 1, wherein the adsorbent material comprises a hydrophilic material.
7. The absorbent article of claim 1, wherein the adsorbent particles are directly affixed to a fiber or a polymeric film.
8. The absorbent article of claim 6, wherein the adsorbent material comprises an inorganic material.

9. The absorbent article of claim 8, wherein the adsorbent material is selected from the group consisting of activated carbon, metal oxides, silicates, zeolites, carbonates, phosphates, borates and aerogels.
10. The absorbent article of claim 6, wherein the adsorbent material comprises an organic material.
11. The absorbent article of claim 10, wherein the adsorbent material is selected from the group consisting of cellulosic materials, starches, chitins, alginates and synthetic polymers.
12. The absorbent article of claim 6, wherein the adsorbent material is a mixture of organic and inorganic materials.
13. The absorbent article of claim 1, wherein the adsorbent material has an interstitial spacing of between about 100 and about 1,000 microns.
14. The absorbent article of claim 1, wherein the adsorbent material has an intraparticle pore size of between about 100 and about 0.2 microns.
15. The absorbent article of claim 1, wherein less than about 2 percent of the pore volume comes from pores less than about 1 micron.
16. An absorbent article comprising a containment means and particles of at least one adsorbent material, while in the containment means about 10 to about 100 percent of the adsorbent particles have a permeability of at least about 1,000 K, the adsorbent particles being present in the containment means in an amount of from about
5 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

17. The absorbent article of claim 16, wherein the adsorbent particles have a permeability of between about 1,000 and about 4,000 K.
18. The absorbent article of claim 16, wherein the containment means comprises a topsheet and a backsheet.
19. The absorbent article of claim 16, wherein the adsorbent material comprises a hydrophilic material.
20. The absorbent article of claim 16, wherein the adsorbent particles are directly affixed to a fiber or a polymeric film.
21. The absorbent article of claim 16, wherein the adsorbent material is a mixture of organic and inorganic materials.
22. The absorbent article of claim 16, wherein the adsorbent material has an interstitial spacing of between about 100 and about 1,000 microns.
23. The absorbent article of claim 16, wherein the adsorbent material has an intraparticle pore size of between about 100 and about 0.2 microns.
24. The absorbent article of claim 16, wherein less than 2 percent of the pore volume comes from pores less than about 1 micron.
25. An absorbent article comprising a containment means and particles of at least one adsorbent material, while in the containment means the adsorbent particles have a complex fluid retention capacity of at least about 2 g/g, the adsorbent particles being present in the containment means in an amount of from about 10 to about 100 percent
5 based on the total weight of the containment means and the adsorbent particles.

26. The absorbent article of claim 25, wherein the adsorbent particles have a complex fluid retention capacity of between about 1 and about 15 g/g.

27. The absorbent article of claim 25, wherein the adsorbent particles have a complex fluid retention capacity of between about 2 and about 8 g/g.

28. The absorbent article of claim 25, wherein the adsorbent particles have a complex fluid retention capacity of between about 2 and about 6 g/g.

29. The absorbent article of claim 25, wherein the containment means comprises a topsheet and a backsheet.

30. The absorbent article of claim 25, wherein the adsorbent material comprises a hydrophilic material.

31. The absorbent article of claim 25, wherein the adsorbent particles are directly affixed to a fiber or a polymeric film.

32. The absorbent article of claim 25, wherein the adsorbent material is a mixture of organic and inorganic materials.

33. The absorbent article of claim 25, wherein the adsorbent material has an interstitial spacing of between about 100 and about 1,000 microns.

34. The absorbent article of claim 25, wherein the adsorbent material has an intraparticle pore size of between about 100 and about 0.2 microns.

35. The absorbent article of claim 25, wherein less than 2 percent of the pore volume comes from pores less than about 1 micron.

36. An absorbent article comprising a containment means and particles of at least one adsorbent material, while in the containment means the adsorbent particles having a minimum average particle size of at least about 200 microns with a standard deviation of at least about 25 percent of the average particle size, the adsorbent particles
5 being present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

37. The absorbent article of claim 36, wherein the adsorbent particles have an average particle size of between about 200 and about 800 microns.

38. The absorbent article of claim 36, wherein the adsorbent particles have an average particle size of between about 300 and about 600 microns.

39. The absorbent article of claim 36, wherein the adsorbent particles have an average particle size of between about 400 and about 500 microns.

40. The absorbent article of claim 36, wherein the containment means comprises a topsheet and a backsheet.

41. The absorbent article of claim 36, wherein the adsorbent material comprises a hydrophilic material.

42. The absorbent article of claim 36, wherein the adsorbent particles are directly affixed to a fiber or a polymeric film.

43. The absorbent article of claim 36, wherein the adsorbent material is a mixture of organic and inorganic materials.

44. The absorbent article of claim 36, wherein the adsorbent material has an interstitial spacing of between about 100 and about 1,000 microns.

45. The absorbent article of claim 36, wherein the adsorbent material has an intraparticle pore size of between about 100 and about 0.2 microns.

46. The absorbent article of claim 36, wherein less than 2 percent of the pore volume comes from pores less than about 1 micron.

47. An absorbent article comprising a containment means and particles of at least one adsorbent material, while in the containment means the adsorbent particles have a multi-modal particle size distribution, the adsorbent particles being present in the containment means in an amount of from about 10 to about 100 percent based on the total weight of the containment means and the adsorbent particles.

48. The absorbent article of claim 47, wherein the containment means comprises a topsheet and a backsheet.

49. The absorbent article of claim 47, wherein the adsorbent material comprises a hydrophilic material.

50. The absorbent article of claim 47, wherein the adsorbent particles are directly affixed to a fiber or a polymeric film.

51. The absorbent article of claim 47, wherein the adsorbent material is a mixture of organic and inorganic materials.

52. The absorbent article of claim 47, wherein the adsorbent material has an interstitial spacing of between about 100 and about 1,000 microns.

53. The absorbent article of claim 47, wherein the adsorbent material has an intraparticle pore size of between about 100 and about 0.2 microns.

54. The absorbent article of claim 47, wherein less than 2 percent of the pore volume comes from pores less than about 1 micron.

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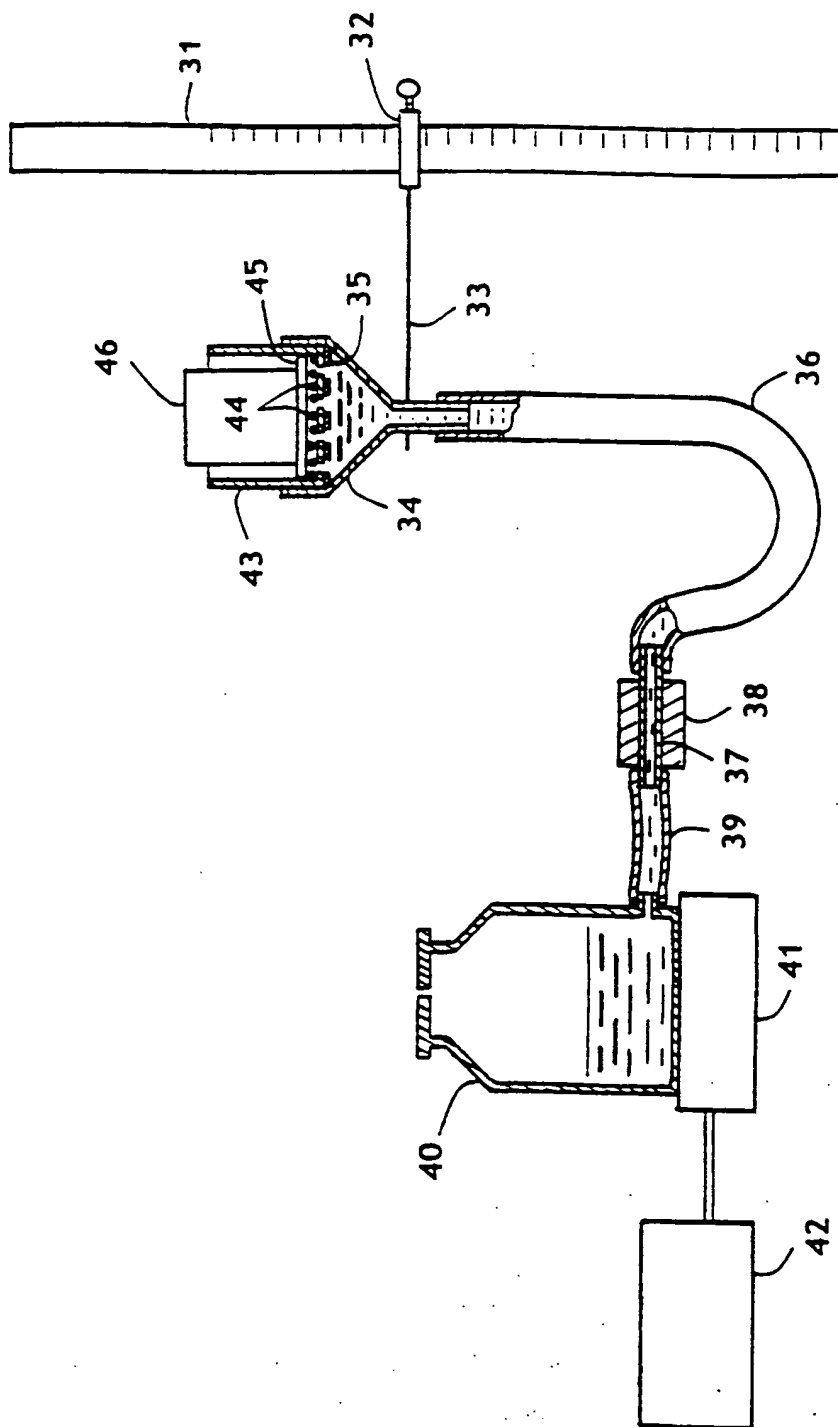


FIG. 1

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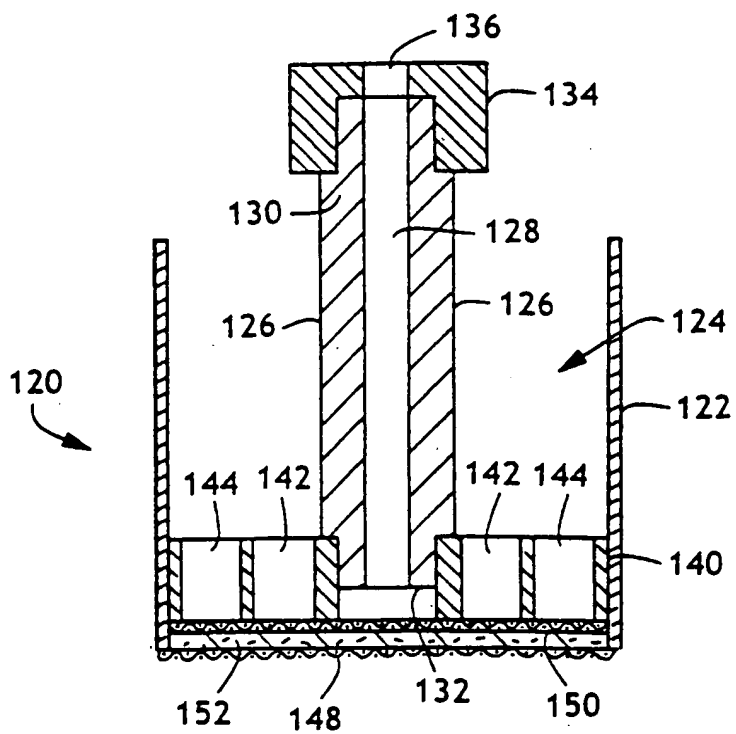


FIG. 2

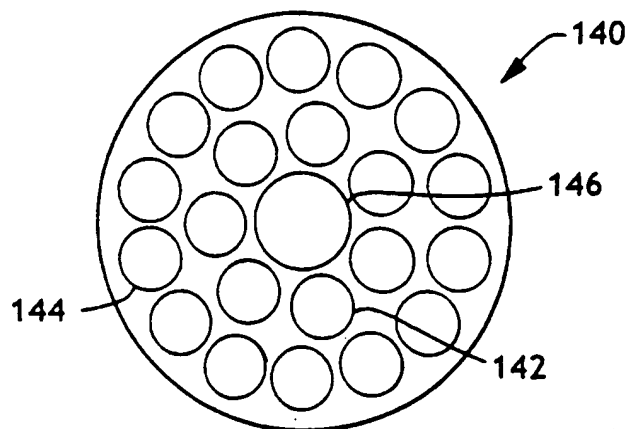


FIG. 3

INTERNATIONAL SEARCH REPORT

Int. Patent Application No.
PCT/US 00/10323

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61L15/18 A61L15/20 A61L15/46 A61L15/60 A61F13/53

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61L A61F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 539 019 A (SUSKIND STUART ET AL) 23 July 1996 (1996-07-23)	1-12, 16, 18-21, 36-43, 47-51
Y	abstract column 5, line 9-47 — -/-	1-16, 18-21, 25-31, 36-43, 47-51

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

4 August 2000

Date of mailing of the international search report

16/08/2000

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Böhm, I

INTERNATIONAL SEARCH REPORT

Int. Patent Application No.

PCT/US 00/10323

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	<p>WO 97 07761 A (KIMBERLY CLARK CO) 6 March 1997 (1997-03-06)</p> <p>abstract page 3, line 20-37 page 6, line 1-35</p>	<p>1-16, 18-21, 25-31, 36-43, 47-51</p>
X	<p>WO 97 27884 A (PEARLSTEIN MELBA & LF) 7 August 1997 (1997-08-07)</p> <p>page 5 page 6, paragraphs 1,2 page 9, paragraphs 1-3 page 10, paragraphs 2,3 claims 20-25,28</p>	<p>1-12,16, 18-21, 36-43, 47-51</p>
X	<p>US 5 419 956 A (ROE DONALD C) 30 May 1995 (1995-05-30)</p> <p>abstract column 3, line 3-26 column 8, line 6-17</p>	<p>1-12,16, 18-21, 36-43, 47-51</p>

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 17

Present claims 1-24 relate to a product defined (inter alia)

by reference to the following parameter (mentioned in claims 1,16,17):
 P1: gel bed permeability 'K!', defined in the description as the following equation:

$$K = Q * (H * \mu) / (A * \rho * P), \text{ with}$$

Q: flow rate

H: height of the bed

μ : liquid viscosity

A: cross-sectional area

ρ : liquid density

P: hydrostatic pressure

The use of these parameters in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search of claims 1-24 has been restricted to those adsorbent particles in absorbent articles in which the particles are that according to claims 2-15, 18-24

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

Int. J. Appl. No.

PCT/US 00/10323

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